



TYPE Surface treatment of PTFE

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0.6 gel calf 62 ug/gel
0.6 water
1.2 heparin
0.1 mL surf MED - diphosphate (SR-1)
- in water.

After treatment, water plated to PTFE
where it was treated with emulsion

preparation of two emulsions C-11-22-96
1. - with 60 ug/mL calf gel
2. with 30 ug/mL calf gel.

(1) All emulsions are marked as E-n

E-1 30 ug/mL 0.6 g gel.
0.6 mL H₂O
1.2 mL Heparin
52 ug surf SR-1

E-2 1.2 g gel 4.6% gel.
1.2 mL Heparin
52 ug SR-1
+ 34 ug SP-1
2.51

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Witnessed & Understood by me,
Doreen Hene

Date

Invented by Argene Parkhurst

Date

Reported by Argene Parkhurst



86

Project No. _____

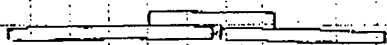
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~~# 2~~ ~~0.4 g/L~~ ~~x .46~~

0.5 mL

~~0.05 g/L~~

Two stripes were treated with Dumbbells
E-2. 5x25 mm and covered with

Collagen gel. (10x5x2 mm)

Lap shear test showed adhesive failure for
one stripe at 86 g (peak value), and
then collagen gel was put into upper fixture
and test was repeated. With this second
stripe we observed cohesive failure at 156 g!

Surprisingly, without pretreatment cohesive failure
occurred for same type of test at 71 g.

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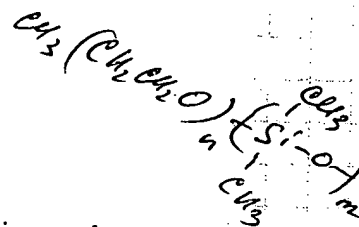
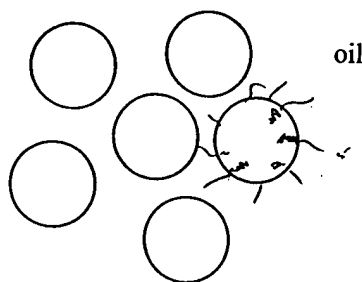
Recorded by

Lupine Pashkov



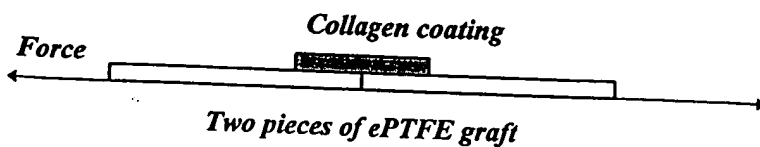
As has been mentioned on pp. 85-86, emulsions can be used to improve adherence to ePTFE. The only possibility to get a good contact between gel and PTFE is to improve penetration of the gel to the pores. It can be done with collagen water-in-oil emulsions.

I have been continuing to work on adhesion problem and I came to conclusion that a new elegant way to improve adherence can be utilized. The heart of the adhesion problem is to get a strong interface between chemically different materials, such as highly hydrophobic PTFE and collagen gel. The only possibility to get good contact is to improve penetration of collagen to highly porous surface of PTFE. It can be done with collagen water-in-oil emulsion. Emulsion of such a kind can be presented as collagen gel droplets sitting in oil:



Those droplets contain concentrated collagen gel with persulfate. Emulsion can be stabilized with non-ionic surfactants. Surface of droplets is hydrophobic and therefore, they can easily penetrate into the pores. The core of droplets is photoreactive collagen gel. Therefore, it can chemically interact with similar gel applied to the surface.

Two different emulsions were prepared. First emulsion (E-1) contained 30mg/mL calf collagen gel + hexane + surfactant. Second emulsion (E-2) was prepared with more concentrated (60 mg/mL) collagen gel. An appropriate amount of sodium persulfate solution was added to each emulsion. To get quantitative data on adherence we used lap shear test:



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Adherence to PTFE

Size of ePTFE stripes was 25x5 mm, and size of collagen coating was 10x5x2 mm. Area of contact between ePTFE and collagen was 5x5 mm². From this experiment, we can obtain value of adhesive energy or work of adhesion (Force/area).

Finally, we have two stripes covered with collagen, which can display different adhesive strength. Therefore, from one experiment, we can obtain two different values for different stripes.

Results for pretreatment with E-2:

For one stripe, adhesive strength was 86 g whereas for the second one we observed cohesive failure at 156 g.

Results for pretreatment with E-1:

untreated ePTFE shows failure at 30 g, whereas treated sample at 58 g (1st stripe) and 75, (2nd stripe).

After failure, same piece of gel was applied to same surfaces of treated ePTFE gels and additionally cured for 15 S (initial curing time is 45 S).

Failure took place at 105 g and 113 g for different stripes.

Conclusion: pretreatment with emulsions containing collagen, persulfate, non-ionic surfactants can be used to improve adherence of collagen to ePTFE. First results show that collagen can penetrate to the pores (after failure we observed some amount of collagen gel on the PTFE surface).

There are several variables that influence the adherence:

1. Emulsion. Viscosity, concentration of collagen, persulfate, and surfactants, as well as chemical structure of surfactants are very important.
2. Curing time. Dependence of adherence on curing time indicates that we have some penetration of collagen to the pores, and/or we observe changes in surface energy of collagen gel with curing. This can be observed visually: undercured gel is "wet" whereas completely cured material is "dry".

Witnessed & Understood by

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Do... of pretreatment in vivo

Adhesion

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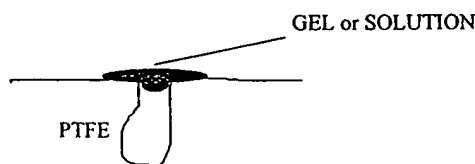
Adhesion of collagen materials to the live tissue and to the PTFE grafts has very

... has a chemical origin. Chemical bonding makes interface bond strong. For the gel/PTFE interface situation is less favorable. The reason for this is that PTFE is one of the most hydrophobic and chemically inert materials. However high porosity of the PTFE material could induce mechanical type of adhesion. In this case penetration of hydrogels through the pores results in high adherence.

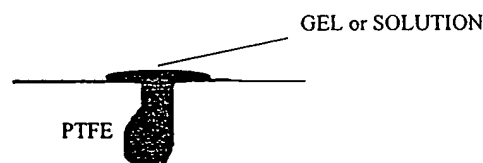
Despite high porosity of this material water (and viscous polymer solutions) will likely not penetrate into the pores because of poor wettability of PTFE surface. Therefore, it is necessary to design experiments for appropriate surface treatment and analysis of gels penetration to the PTFE pores.

There are two extreme variants of interaction of porous material with water or solution of water soluble polymers:

A. Poor wettability



B. Good wettability



Inventors' Signatures:

Read, Understood, and Witnesses by:

There are different ways to improve wettability of hydrophobic surfaces, for instance, plasma treatment, high energy laser irradiation, etching using strong acids etc. From the point of view of product application, however, only a few methods are acceptable.

Wettability of PTFE can be improved using surfactants which represent molecules having hydrophobic and hydrophilic fragments. We are going to use non-ionic surfactants or block copolymers such as

- Poly(propylene oxide)-block-poly(ethylene oxide)-block-Poly(propylene oxide)
- Poly(ethylene oxide)-block-poly(dimethylsiloxane)
- Stearate-block-poly(ethylene oxide)-block-Stearate

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